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First-Order Parametric Model of Reflectance Spectra for Dyed Fabrics

D. AIKEN
S. RAMSEY
T. MAYO

*Signature Technology Office
Tactical Electronic Warfare Division*

S.G. LAMBRAKOS

*Center for Computational Materials Science
Materials Science and Technology Division*

J. PEAK

*Signature Technology Office
Tactical Electronic Warfare Division*

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14. ABSTRACT This report describes a first-order parametric model of diffuse reflectance for the purpose of simulating the spectral response of military textiles containing different types and concentrations of near-infrared (NIR) absorbing dyes. This model should contribute to the development of prediction tools, which when given the constituent materials available, will provide for the design of optimal composite systems, i.e. military combat uniforms allowing individuals to match the environmental spectral response in the visual, near-infrared, and shortwave-infrared spectral bands. The dyes considered for prototype analysis using this model are triarylamine, tetraaryldiamine, transition metal dithiolene, and indolium iodide. The fabrics considered are camouflage textiles characterized by color variations. The results of this study provide validation of the constructed parametric model, within reasonable error tolerances for practical applications, including NIR spectral characteristics in camouflage textiles, for purposes of simulating NIR spectra corresponding to various dye concentrations in host fabrics, as well as potentially to mixtures of dyes.					
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First-Order Parametric Model of Reflectance Spectra for Dyed Fabrics

Introduction

Parametric models of NIR reflectivity spectra for dyed fabrics are presented, which provides for both their inverse and direct modeling¹. The dyes considered contain spectral features that are of interest to the U.S. Navy for the purpose of simulating NIR spectra corresponding to various dyes and mixtures of dyes in camouflage textiles. The results provide validation of the constructed parametric models, within reasonable error tolerances, and will contribute to the development of a parameter space for process optimization of dyed fabrics. Given the fabric and dyes available, an optimization algorithm could be developed that predicts the optimal dying process and fabric/dye combinations needed to provide multi-spectral concealment for a given environment. To reach the point of process optimization, different absorbing dyes, fabrics, and parametric reflectance models must first be examined. The theory underlying the parametric model, as well as the dyes and fabric chosen for this analysis, are described in subsequent sections. The formulation of the parametric model is based on the inverse analysis approach¹.

Materials and Methods

Dyes. The reflectance characteristics of 4 dyes were examined. Each dye had a wavelength of maximum absorption located in either the near or short-wave infrared bands, with appreciable spectral features extending into the visible spectrum² (Table 1). The dyes were analyzed individually, but have potential to be used in combinations to create custom spectral responses, i.e. reflectance or transmission.

Fabric. The base fabric considered for dye application was a 50% Nylon, 50% Cotton blend Ripstop fabric, i.e. 50/50 NYCO Ripstop. Ripstop fabrics are woven fabrics with reinforcement threads interwoven at regular intervals in a crosshatch pattern³. The fabric was characterized as having a rough surface, finite and non-uniform thickness, heterogeneous composition, and a four color, AOR1 desert camouflage pattern design (Figure 1).

Dyed-Fabric Sample Preparation. For each dye and corresponding solvent (Table 1), a stock solution of 30 g L⁻¹ was made. Serial dilutions of each stock solution were prepared, consisting of 3.0, 1.5, 0.3, 0.15 and 0.03 g L⁻¹. Twenty dye-solutions were prepared in total. One square yard of 50/50 NYCO Ripstop fabric was cut from the roll and placed under a Freeman Schwabe cutting press. A 2"x2" Fremont steel rule cutting die was used in combination with the mechanical press to make 20, 2"x2" fabric samples. Dye-fabric samples were prepared by pipetting 1 mL of each dye solution onto the center of each 2"x 2" fabric piece. The pipetting speed was adjusted as to avoid pooling of the dye solution on the fabric surface. Dye-fabric samples were given 24 hours to dry before total reflectance measurements were made.

Spectral Measurements of Dyed-Fabric Sample. Measurements of total reflectance were made using a UV/Vis/NIR/SWIR spectrophotometer (Perkin-Elmer® LambdaTM 1050, Waltham, MA, USA) in combination with UV WinLab (V6.0.3, Perkin Elmer) software. Reflectance was measured from 250 nm to 2500 nm at 5 nm stepping increments. All measurements were 8

degree hemispherical in total reflectance collection mode. The Lambda 1050 incorporated a double beam, 150 mm integrating sphere housing a photomultiplier tube (PMT) detector for the UV-Vis (175 – 860 nm) region and an Indium Gallium Arsenide (InGaAs) detector for the NIR-SWIR (860 - 2500 nm) region. Radiation sources included a deuterium lamp for operation in the UV (175 – 319 nm) and a tungsten halogen lamp for use in the Vis-NIR-SWIR (319 – 3000 nm). A light source change at 319.2 nm and a detector change at 860.8 nm occurred automatically during monochromator slewing. For UV-Vis wavelengths, the slit width was fixed at 4 nm. For NIR-SWIR wavelengths, the slit width was set to “Servo” mode, which automatically adjusted the slit width during scanning to maintain constant energy at the detector.

Parametric Model of NIR Reflectance Spectra for Dyed Fabrics

Physically consistent parametric models provide a means for investigating the spectral response of a given system, consisting of a given material or combination of materials, and geometry, in terms of both inverse and direct analysis [4,5,6,7]. For reflection from a dyed fabric of finite and nonuniform thickness, whose material composition is heterogeneous, a general framework for parametric modeling of reflectivity spectra can be in terms of formulations based on the Beer-Lambert Law or its approximation, which is adopted for the parametric model considered here [8,9]. Accordingly, a parametric model based on characteristic trend features of reflectance spectra from fabrics, which assumes a first-order absorption process within an average penetration depth, is

$$R_{f+d,M}(\lambda) = (1 - f(\lambda))R_f(\lambda) \quad (1)$$

where

$$f(\lambda) = \sum_{j=1}^N A_j \alpha_j(\lambda) \quad (2)$$

and

$$Z_R = \sum_{n=1}^N w_n \left(R_{f+d,M}(\lambda_n) - R_m(\lambda_n) \right)^2 \quad (3)$$

where α_j is the absorption coefficient of component j . The formal procedure underlying inverse analysis based on Eqs.(1)-(3) entails adjustment of the absorption coefficients defined over the entire wavelength region, if assumed unknown, and the coefficients A_j . This approach defines an optimization procedure where the reflectivity spanning the range of wavelengths is adopted as the quantity to be optimized. Constraint conditions are imposed on the reflectivity by minimizing the objective functions defined by Eq.(3), respectively, where $R_m(\lambda_n)$ is the measured or target reflectivity for wavelength λ_n . The quantities $w_n (n=1, \dots, N)$ are weight coefficients that specify relative levels of influence associated with constraint conditions $R_m(\lambda_n)$.

The mathematical foundation of inverse analysis based on Eqs.(1)-(3) is that of least-squares parameter optimization [10,11]. A significant aspect of least-squares parameter optimization is the choice of a sufficiently complete set of basis functions. This implies that all possible modes of a given process can be modeled parametrically by linear combinations of these functions. Accordingly, the parametric models defined by Eqs. (1)-(3) adopt absorption

coefficients as basis functions. The general form of Eqs. (1) and (2) are based on trend features characteristic of reflectance spectra. Accordingly, the parametric model (Eqs.(1)-(2)) is based on a first-order extinction model of reflectance, whose general form is consistent with spectral features of diffuse reflectance. A derivation of Eq.(1) follows from the first-order relation for extinction

$$\frac{R_f - R_{f+d}}{R_f} \propto \sum_{j=1}^{N_j} \alpha_{ext}(\lambda) \quad (4a)$$

where $\alpha_{ext}(\lambda) = N(C_{abs} + C_{scat}) \quad (4b)$

and N , C_{abs} , C_{scat} are the particle density, absorption and scattering cross sections, respectively, with the conditions that

$$R_d = \frac{R_{f+d}}{R_f} \quad (5)$$

and $\alpha_{ext}(\lambda) \propto \sum_{j=1}^{N_j} C_j \alpha_j(\lambda) \quad (6)$

where R_f , R_d and R_{f+d} are the reflectivities of the base fabric, pure dye and dyed fabric, respectively, and C_j and α_j are the concentrations and absorption coefficients of the j th dye. Equation (5) follows from the assumption that the contribution to the reflectivity due to the fabric can be factored out from that of the combined system of dye and fabric. The proportionality condition given by Eq.(6) represents an important aspect of the constructed parametric model. This condition is necessary in that the extinction coefficient Eq.(4b) is based on absorption and scattering coefficients, C_{abs} and C_{scat} , respectively, with respect to transmission through a finite thickness of particulate material, and thus does not provide for a well defined representation of extinction resulting from both transmission and reflection.

Results and Discussion

A parametric model was examined to characterize the diffuse reflectance properties of dyed fabrics. The model was constructed using a first-order relation for extinction Eq.(4a). Consistent with this relation, diffusely reflected radiation is related to changes in scattering, absorption, and thickness of a dyed fabric layer. The layer is assumed infinitely thick, leaving reflectance in terms of the relative proportions between absorption and scattering. Due to the complex nature of scattering, and the uncertainty in microstructure and dye molecule orientation, the combined influence of absorption and scattering are represented phenomenologically, and thus not associated directly with any physical processes. Accordingly, the function $f(\lambda)$ defined by Eq.(2) represents the combined influence of all underlying physical processes contributing to reflectance within the dyed fabric layer, i.e., reflection, refraction, diffraction, absorption. The function $f(\lambda)$ is a lumped parametric representation consisting of a known absorption coefficient, α , intrinsic to each dye and a parameter A . The absorption coefficient is determined through

inverse analysis of transmission spectra for the dye in solution. The parameter A is adjusted with respect to Eq.(3), i.e., objective function minimization, and accounts for statistical variations in fabric thickness, dye distribution in fabric, and variations in concentration. The $R_f(\lambda)$ denotes the reflectance of the untreated fabric. It is important to note that the absorption coefficient, α , is a known material property as opposed to a phenomenological parameter, i.e., A .

Figures (2)-(5) show reflectance spectra of the four dyes under analysis (Table 1) as a function of dye concentration and wavelength. The reflectance spectrum of a control fabric containing no dye is shown in each figure for comparison. Each experimental spectrum was modeled by adjusting parameter A until the objective function (Eq. (3)) was minimized. Figures (6)-(9) show comparisons between modeled and experimental data for each dye concentration. The good consistency of these fits demonstrates the acceptable prediction accuracy of the parametric model for diffuse reflectance by the dyed fabrics considered. The results also show that absorption coefficients for dyes, which have been determined by inverse analysis of transmission spectra for dyes in solvents, can be validated as reasonable estimates of absorption coefficients for dyes in fabrics. References [12,13] describe the inverse analysis procedure for determination of the dye absorption coefficients considered in this study. Supplementary information concerning the estimated dielectric properties of these dyes as determined by inverse analysis is given in the appendix.

Conclusion

The fitting of modeled to experimental reflectance spectra shown in Figures (6)-(9), as well as the relatively good consistency of these fits, establishes proof of concept for constructing mappings from a process-parameter space, i.e., dye concentrations, to a model-parameter space, i.e., parameters A_j for reflectance spectra. This in turn establishes that the parametric model defined by Eqs. (1) and (2) can be adopted for prediction of dyed fabric reflectance characteristics for process parameters that can be mapped into a physically consistent range of model parameters.

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Table 1: NIR and SWIR Absorbing Dyes

Dye Name	Absorption Max (nm)	Solvent
Triarylamine	978	Acetone
Tetraaryldiamine	949	Acetone
Metal Dithiolene	836	Methylene Chloride
Indolium Iodide	775	Methanol

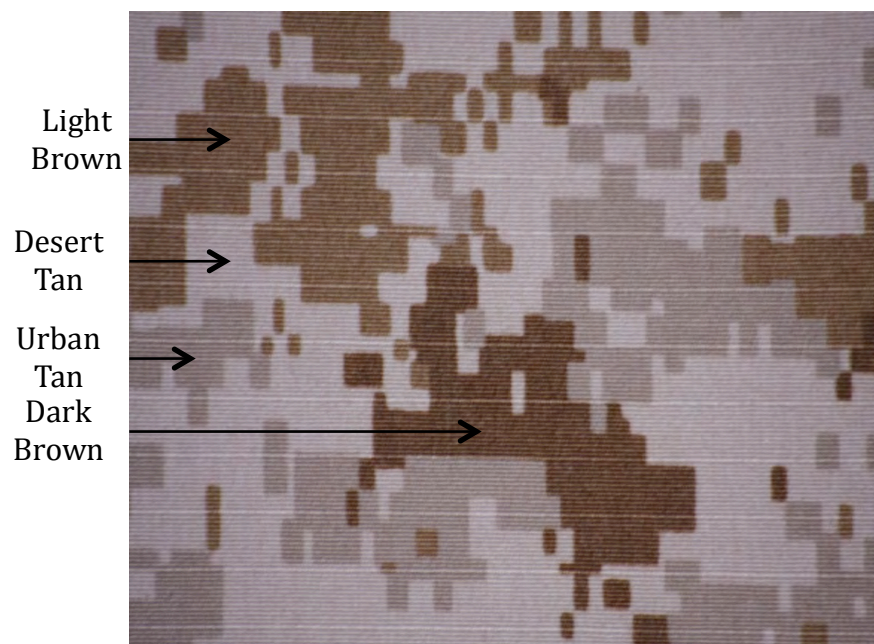


Figure 1. 50/50 NYCO Ripstop fabric considered for dye application. Fabric made of 50% Nylon / 50% Cotton blend.

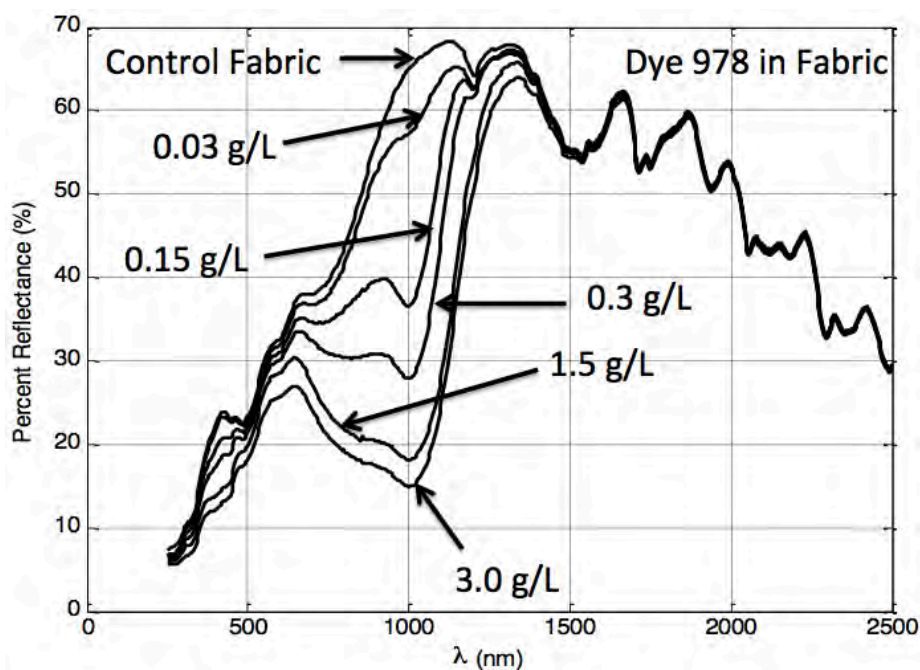


Figure 2. Experimentally measured reflectance spectra for fabric samples containing various concentrations of triarylamine dye as a function of wavelength. A control spectrum for fabric without dye is shown for comparison.

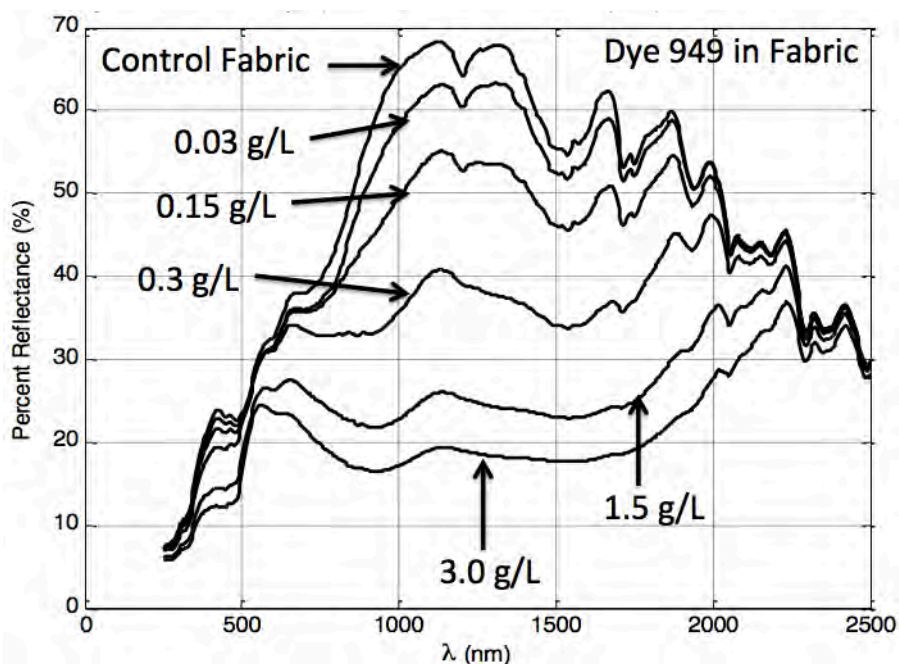


Figure 3. Experimentally measured reflectance spectra for fabric samples containing various concentrations of tetraaryldiamine dye as a function of wavelength. A control spectrum for fabric without dye is shown for comparison.

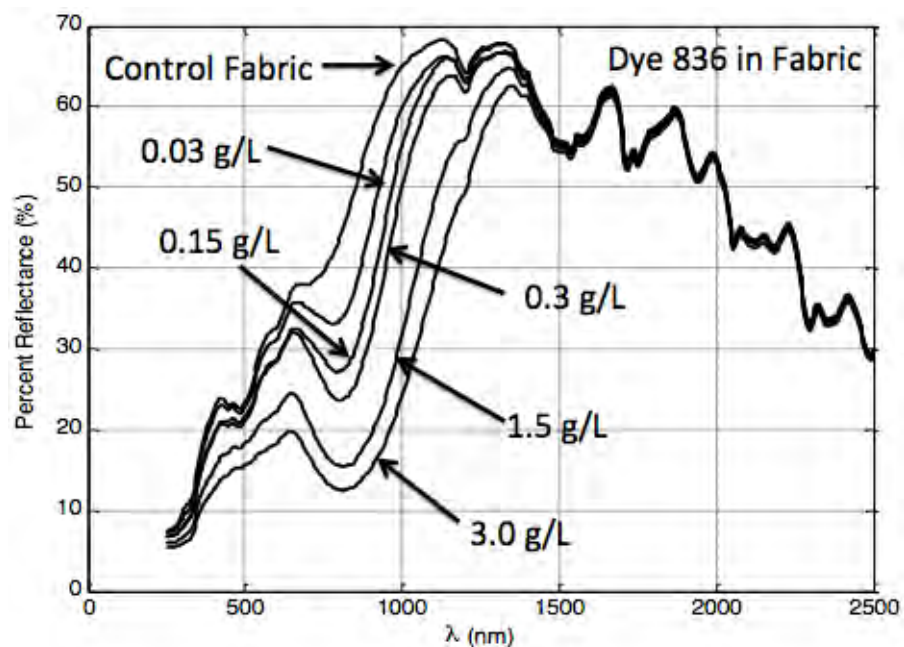


Figure 4. Experimentally measured reflectance spectra for fabric samples containing various concentrations of transition metal dithiolene dye as a function of wavelength. A control spectrum for fabric without dye is shown for comparison.

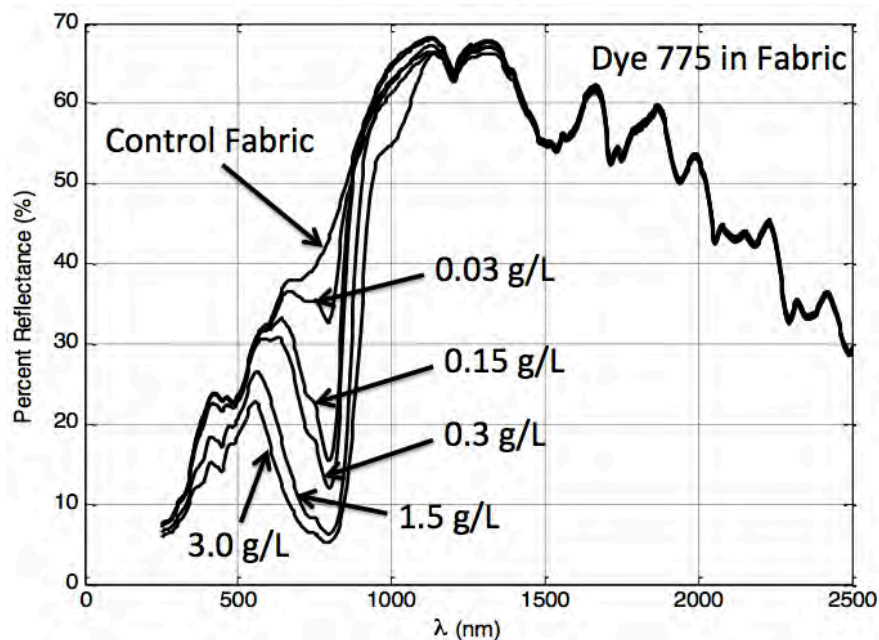
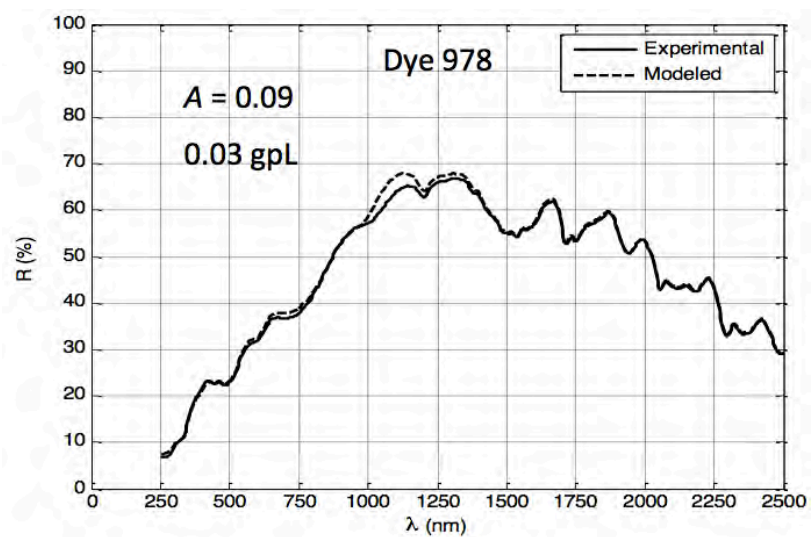
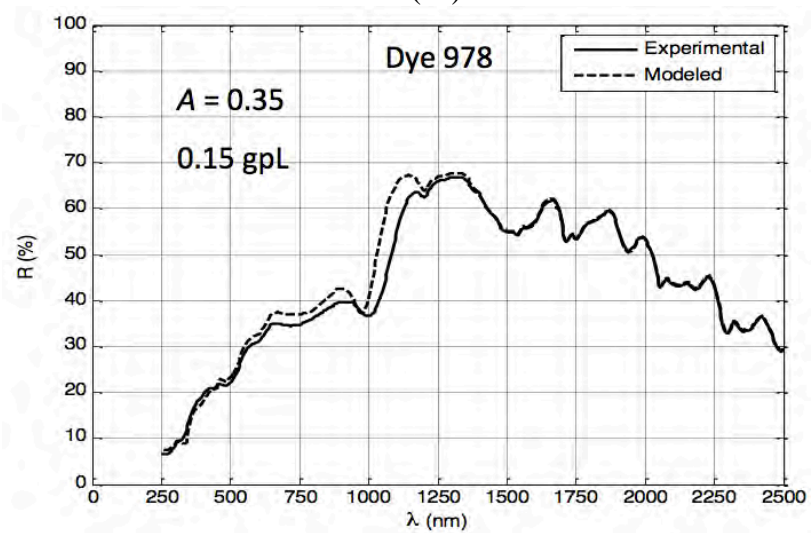


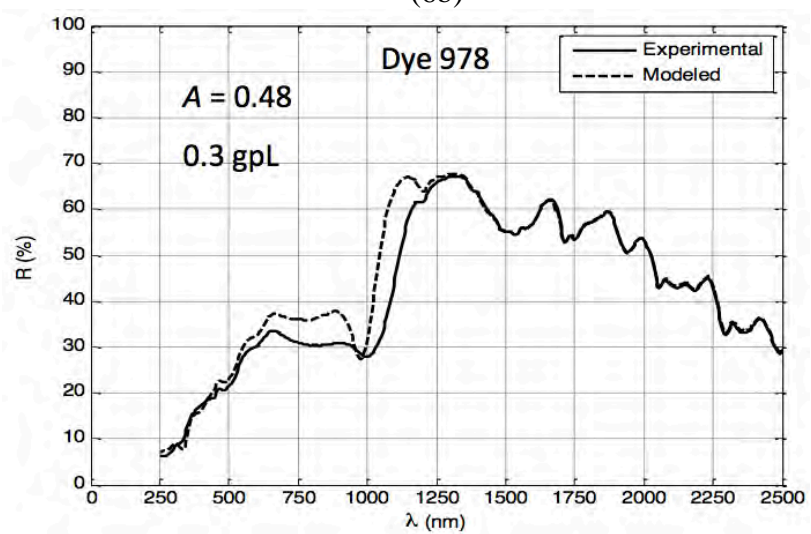
Figure 5. Experimentally measured reflectance spectra for fabric samples containing various concentrations of indolium iodide dye as a function of wavelength. A control spectrum for fabric without dye is shown for comparison.



(6a)



(6b)



(6c)

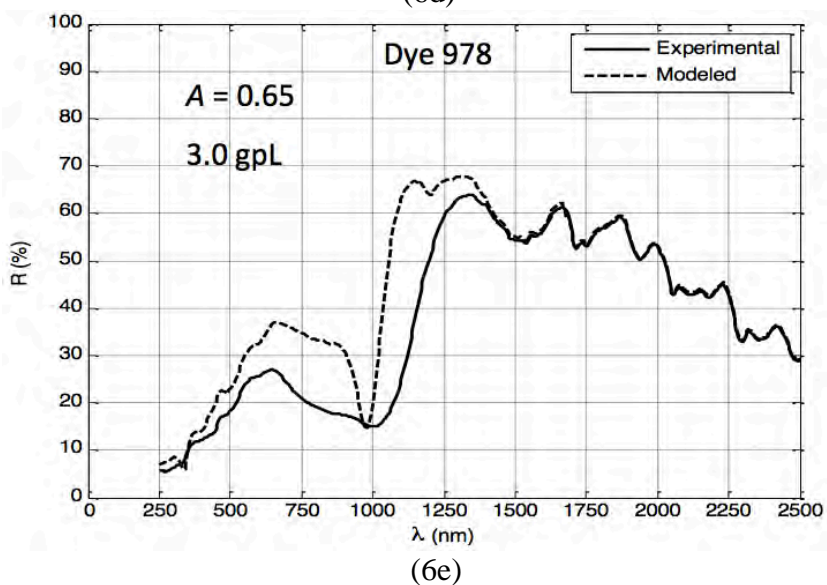
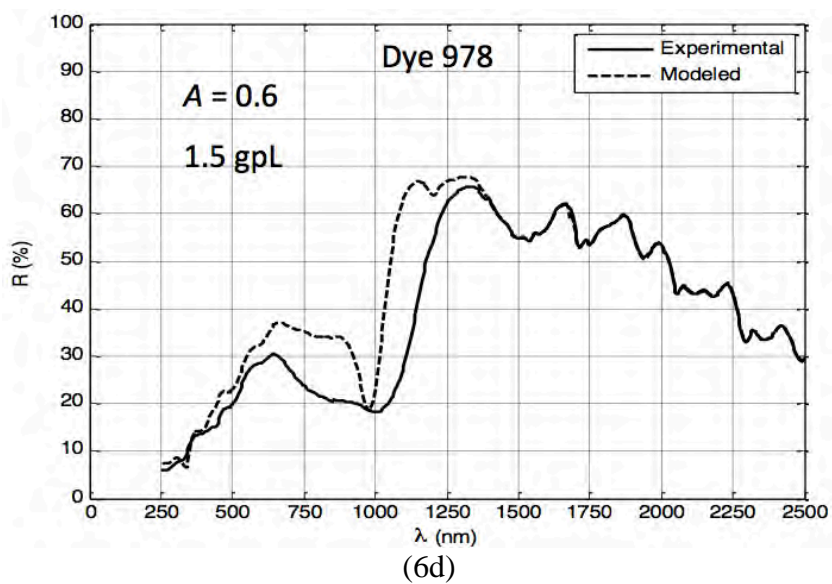
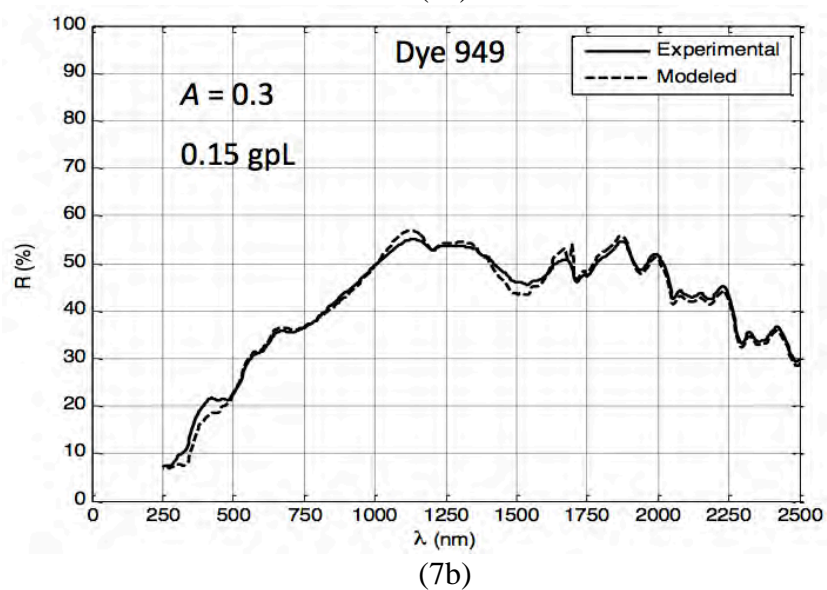
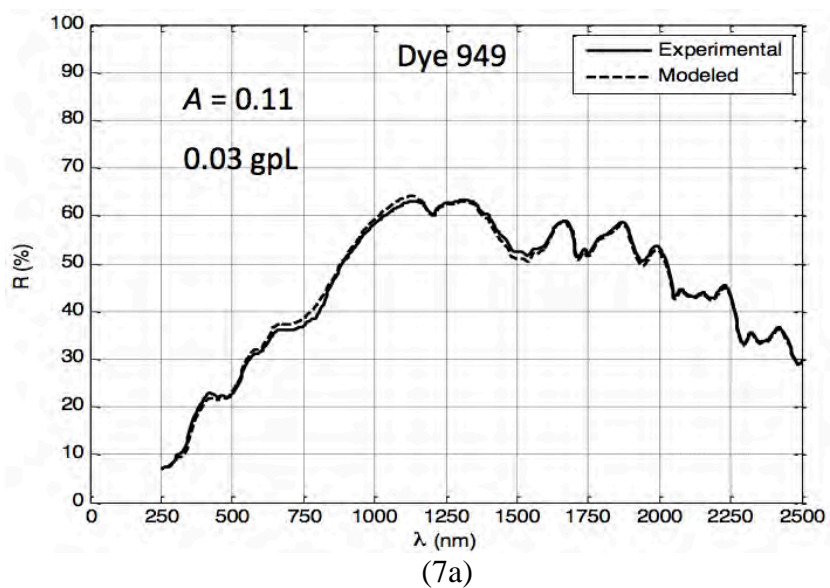
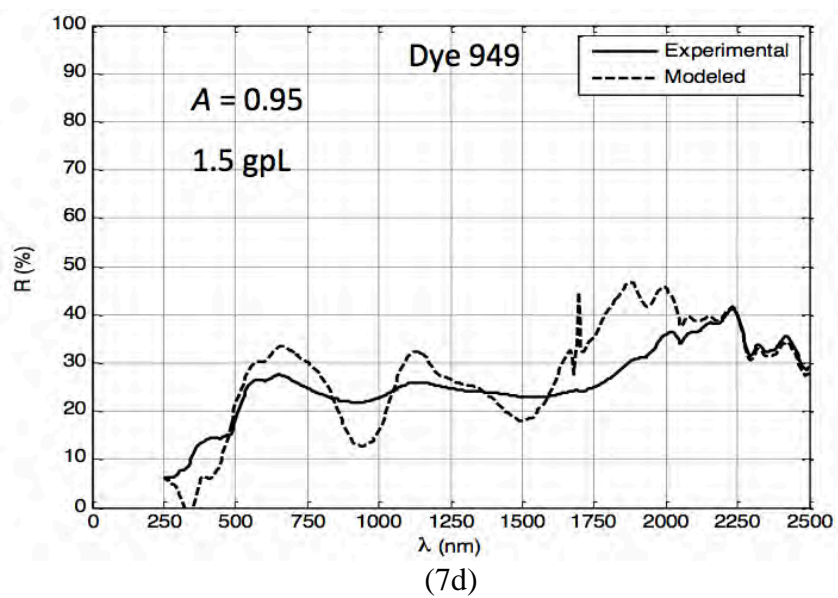
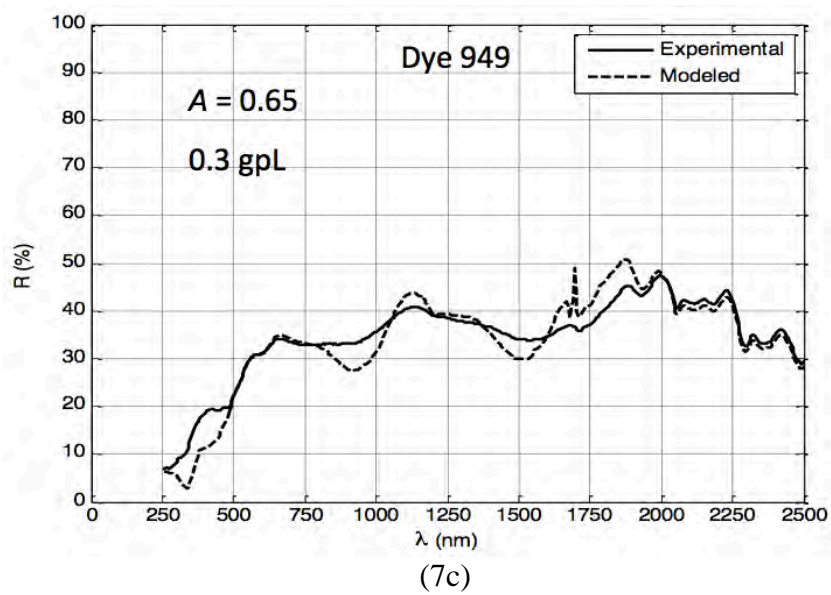


Figure 6. Modeled versus measured reflectance spectra for fabrics containing various concentrations of triarylamine dye as a function of wavelength.





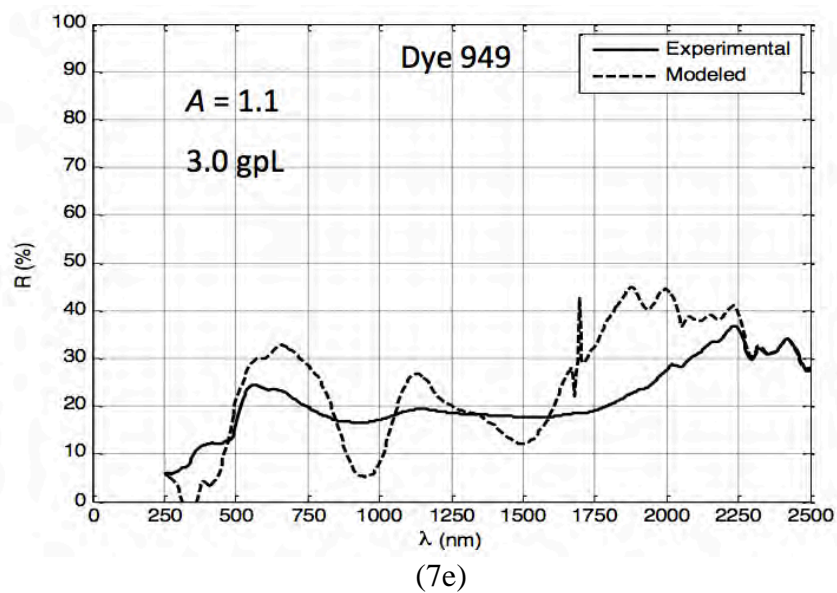
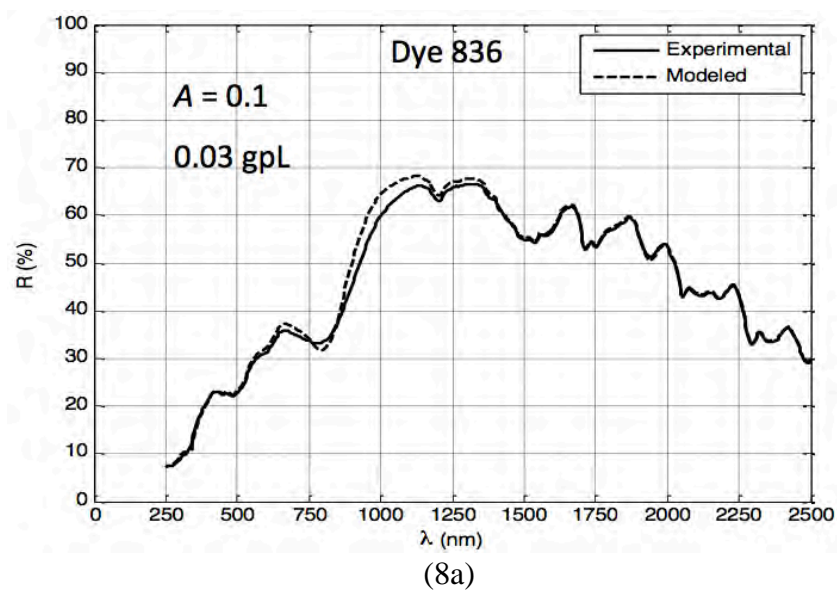
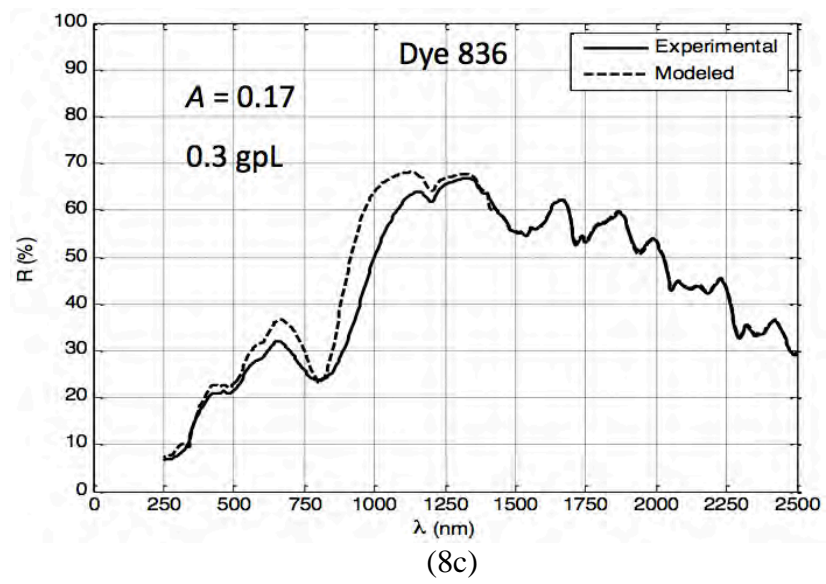
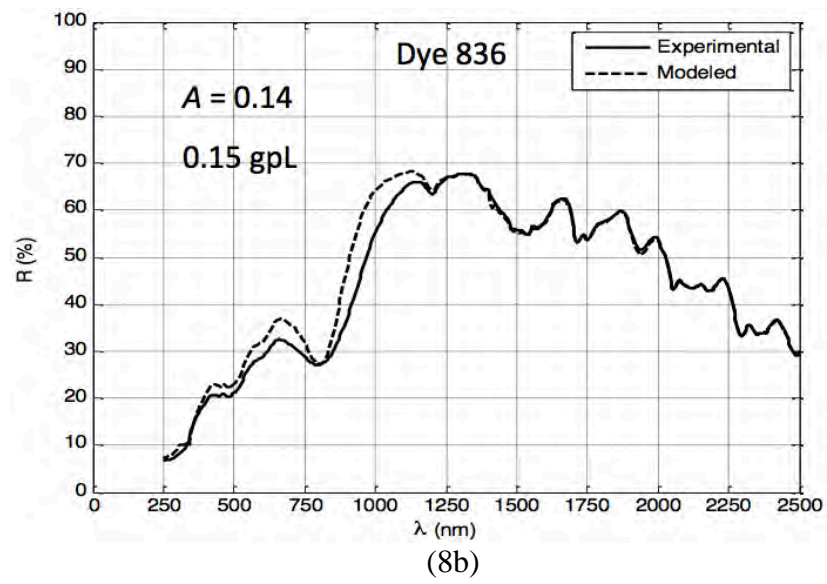


Figure 7. Modeled versus measured reflectance spectra for fabrics containing various concentrations of tetraaryldiamine dye as a function of wavelength.





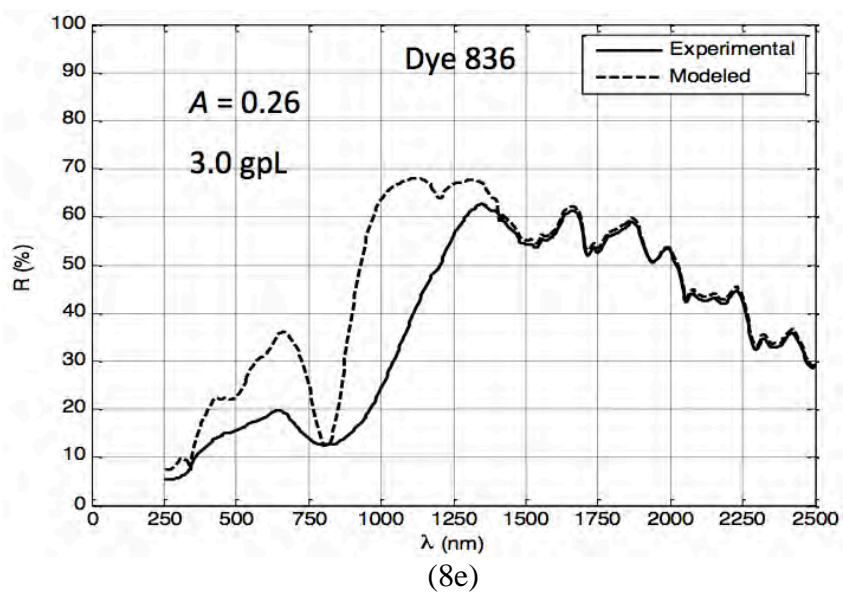
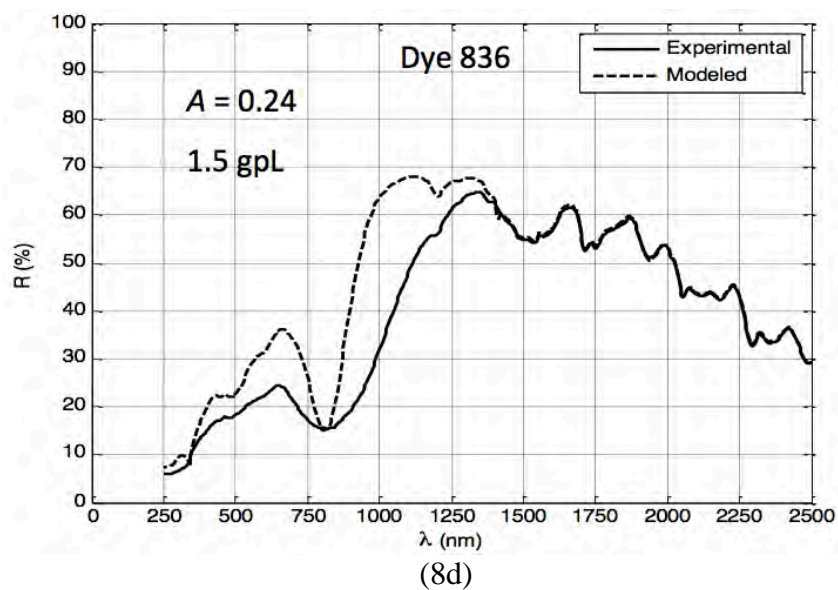
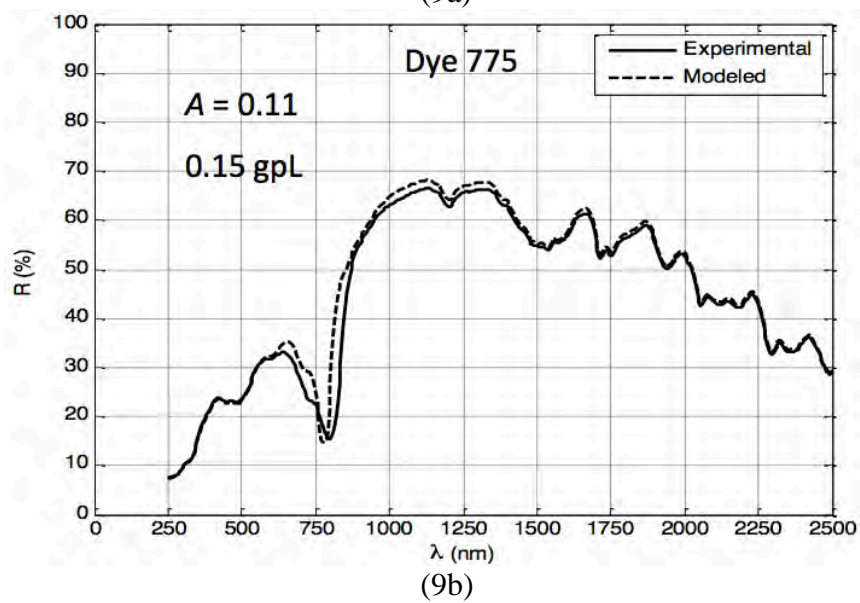
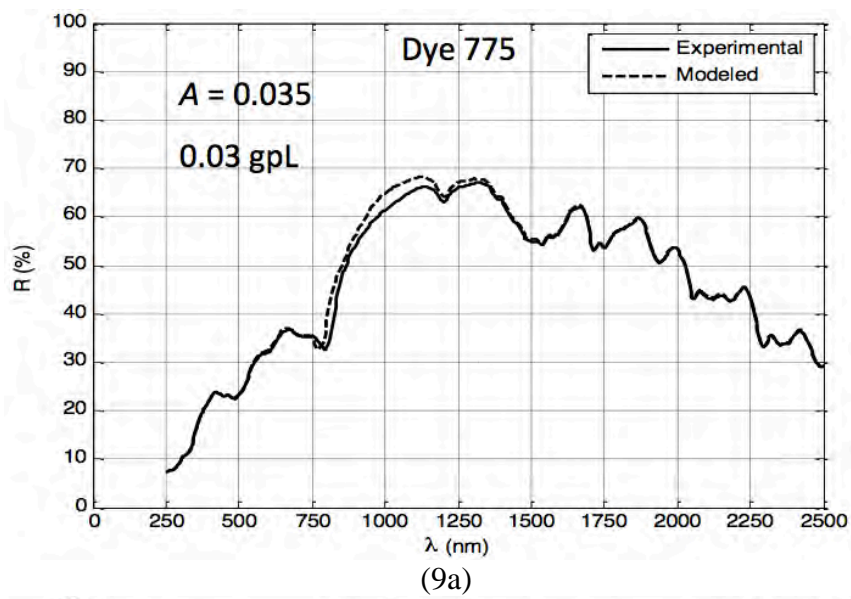
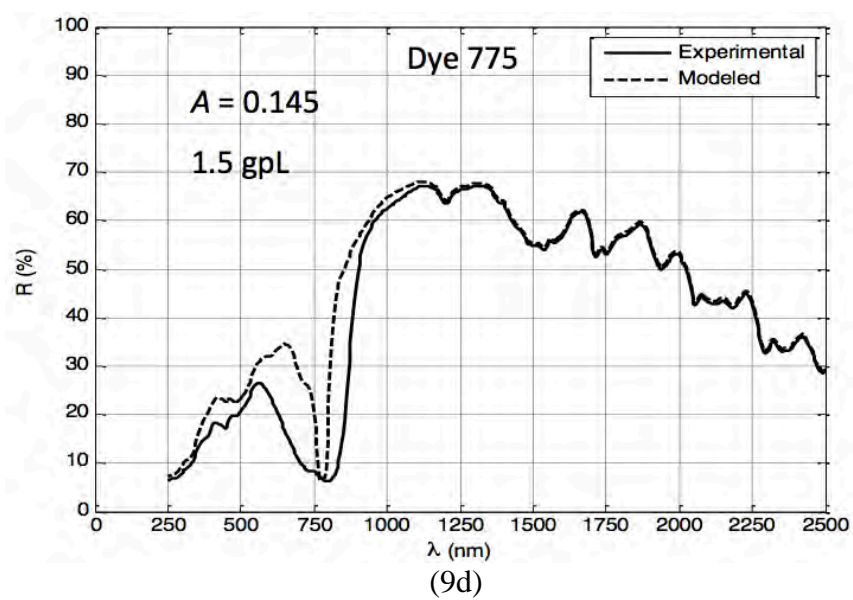
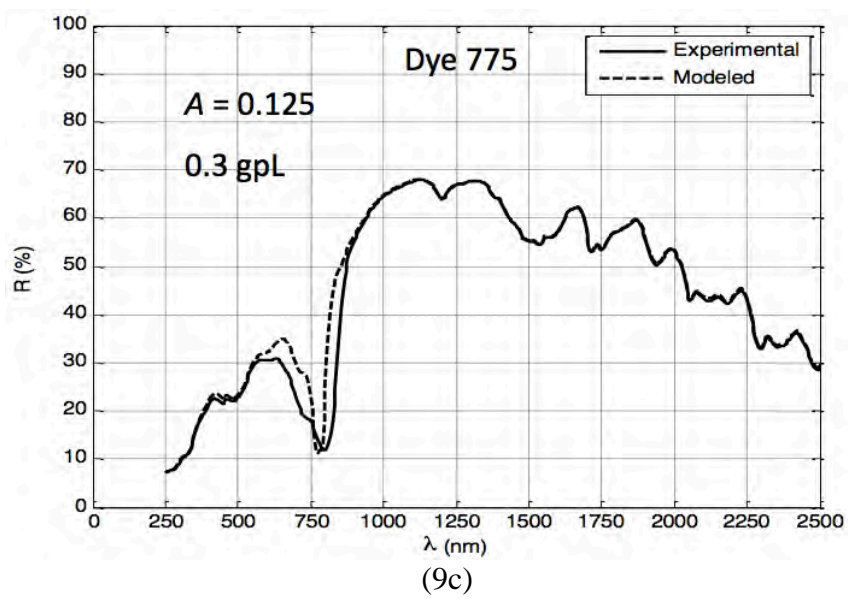


Figure 8. Modeled versus measured reflectance spectra for fabrics containing various concentrations of transition metal dithiolene dye as a function of wavelength.





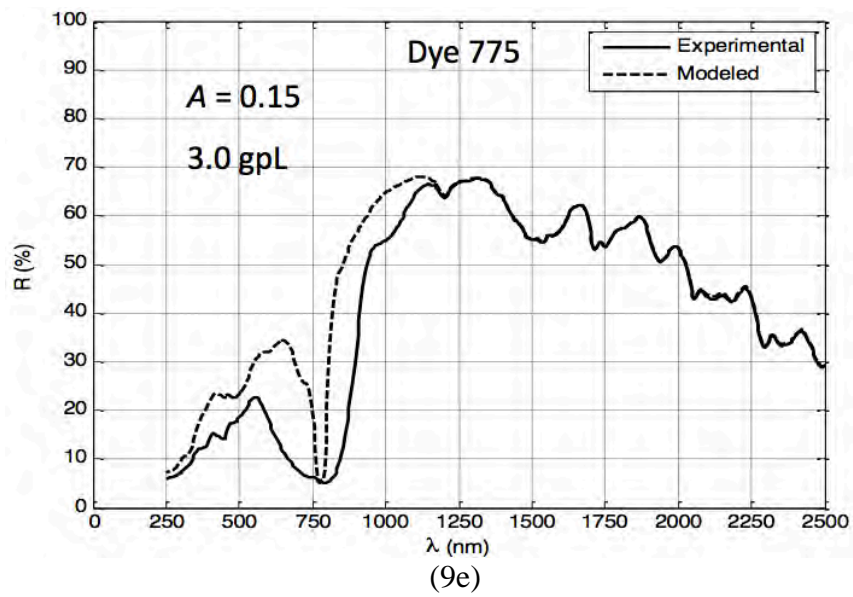


Figure 9. Modeled versus measured reflectance spectra for fabrics containing various concentrations of indolium iodide dye as a function of wavelength.

APPENDIX

Dielectric Response Functions for Dyes Obtained by Inverse Analysis

Supplementary information is given here for the inverse analyses described in references 12 and 13, which are for determination of estimated dielectric response properties of triarylamine (Dye 978), tetraaryldiamine (Dye 949), transition metal dithiolene (Dye 836) and indolium iodide (Dye 775) [2]. For transmission through a sample of finite thickness, a consistent parametric representation of the transmissivity function is given by

$$T(\lambda) = \frac{(1 - R)^2}{R^2 \exp(-\alpha d) + \exp(\alpha d) - 2R \cos \xi} \quad (7)$$

$$R = \frac{(n - 1)^2 + k^2}{(n + 1)^2 + k^2} \quad (8)$$

$$\alpha = \frac{4\pi k}{\lambda} \quad (9)$$

$$\xi = \frac{4\pi n d}{\lambda} \quad (10)$$

$$n = \frac{1}{\sqrt{2}} \left[\sqrt{\varepsilon_r^2 + \varepsilon_i^2} + \varepsilon_r \right]^{1/2} \quad (11)$$

$$k = \frac{1}{\sqrt{2}} \left[\sqrt{\varepsilon_r^2 + \varepsilon_i^2} - \varepsilon_r \right]^{1/2} \quad (12)$$

$$T(\lambda_n) = T_C(\lambda_n) \quad (13)$$

$$\varepsilon_r(\lambda) = \sum_{l=1}^N C_{rl} f_{rl}(\lambda) \quad \text{and} \quad \varepsilon_i(\lambda) = \sum_{l=1}^N C_{il} f_{il}(\lambda) \quad (14)$$

where f_{rl} , f_{il} and C_{rl} , C_{il} represent an optimal set of basis functions and their weighting coefficients, respectively, for a given set of transmission-spectra measurements. The basis functions describe the permittivity function within the region bounded by wavelengths λ_{min} and λ_{max} , respectively. The wavelengths λ_n and quantities T_C specify constraint conditions on the transmissivity. For parametric representation of the sample's dielectric response, a sufficiently optimal set of basis functions include Lorentz oscillators, as well as other functions satisfying the Kramer-Kronig relations [7].

The formal procedure underlying the inverse method considered here [12,13] entails the adjustment of the permittivity function defined over the entire wavelength region. This approach defines an optimization procedure where the transmission spanning the range of wavelengths is adopted as the quantity to be optimized. The constraint conditions are imposed on the transmission by minimizing the objective function defined by

$$Z_T = \sum_{n=1}^N w_n \left(T(\lambda_n) - T_m(\lambda_n) \right)^2 \quad (15)$$

where $T_m(\lambda_n)$ is the measured or target transmission for wavelength λ_n . The quantities w_n ($n=1, \dots, N$) are weight coefficients that specify relative levels of influence associated with constraint conditions $T_m(\lambda_n)$. A consistent parametric representation (Eq.(14)) of the permittivity function $\varepsilon_s(\lambda) = \varepsilon_{sr}(\lambda) + i\varepsilon_{si}(\lambda)$ is that of a linear combination of Lorentz oscillators, i.e.,

$$\varepsilon_{sr} = 1 + \chi_{sr} = 1 + \sum_{j=1}^N \chi_r(\lambda, A_j, g_j, \lambda_{0j}) \quad (16)$$

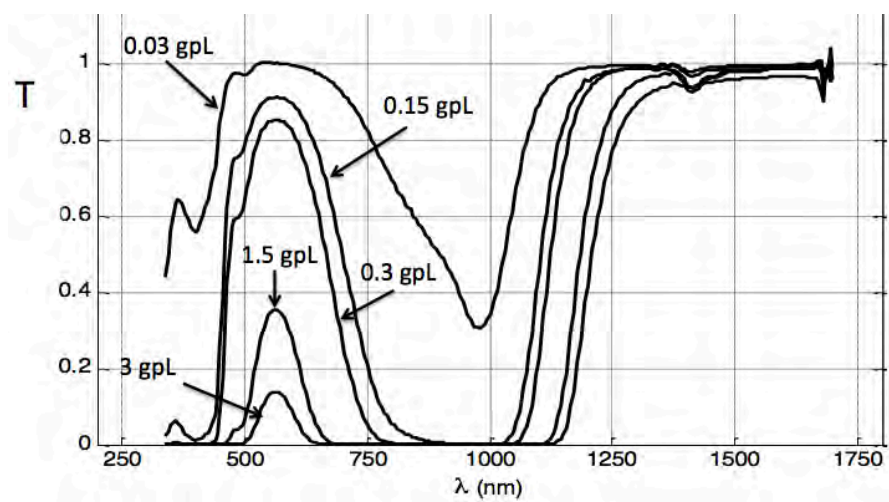
$$\varepsilon_{si} = \chi_{si} = \sum_{j=1}^N \chi_i(\lambda, A_j, g_j, \lambda_{0j}) \quad (17)$$

where

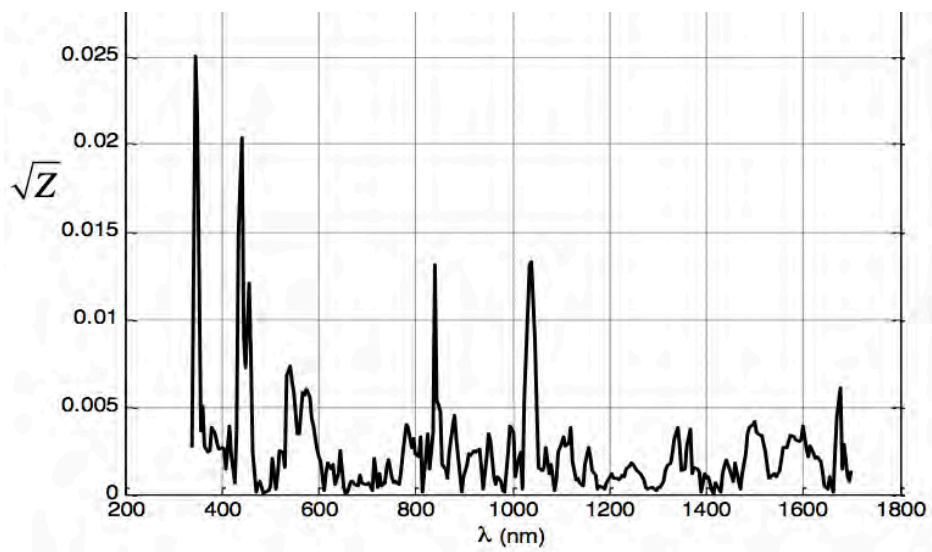
$$\chi_r(\lambda, A, g, \lambda_0) = \frac{A \cdot \lambda^2}{\left(\lambda^2 - \lambda_0^2 \right) + \left(\frac{g \cdot \lambda^2}{\lambda^2 - \lambda_0^2} \right)} \quad (18)$$

$$\chi_i(\lambda, A, g, \lambda_0) = \frac{A \cdot \sqrt{g} \cdot \lambda^3}{\left(\lambda^2 - \lambda_0^2 \right) + g \cdot \lambda^2} \quad (19)$$

and the quantities χ_{sr} and χ_{si} are the real and imaginary parts of the susceptibility function. The procedure for inverse analysis defined by Eqs. (7) through (18) entails adjustment of the parameters A_j , g_j and λ_{0j} defined over the entire wavelength range of interest. This procedure, by inclusion of Eq.(15), is based on least-squares parameter optimization.



(10a)



(10b)

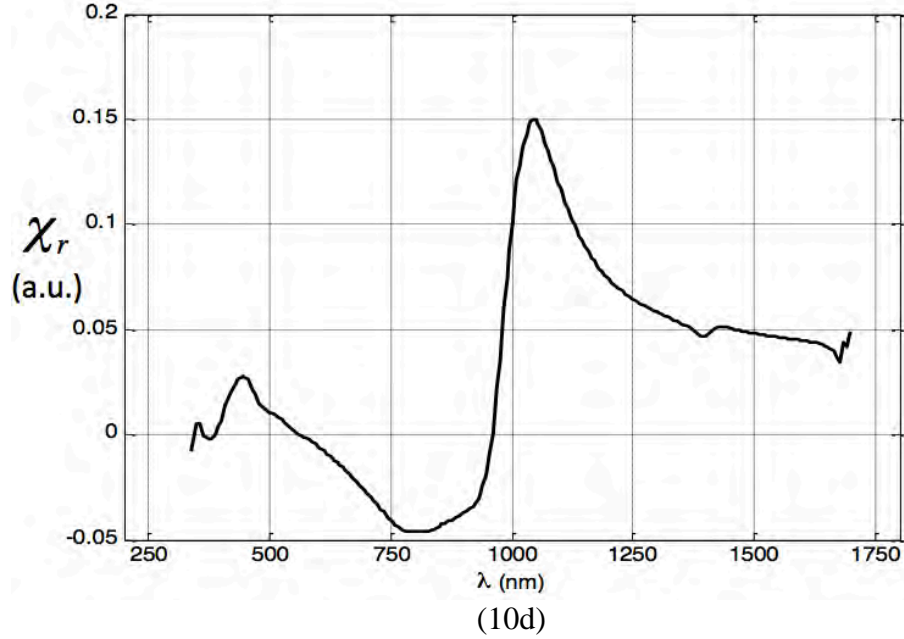
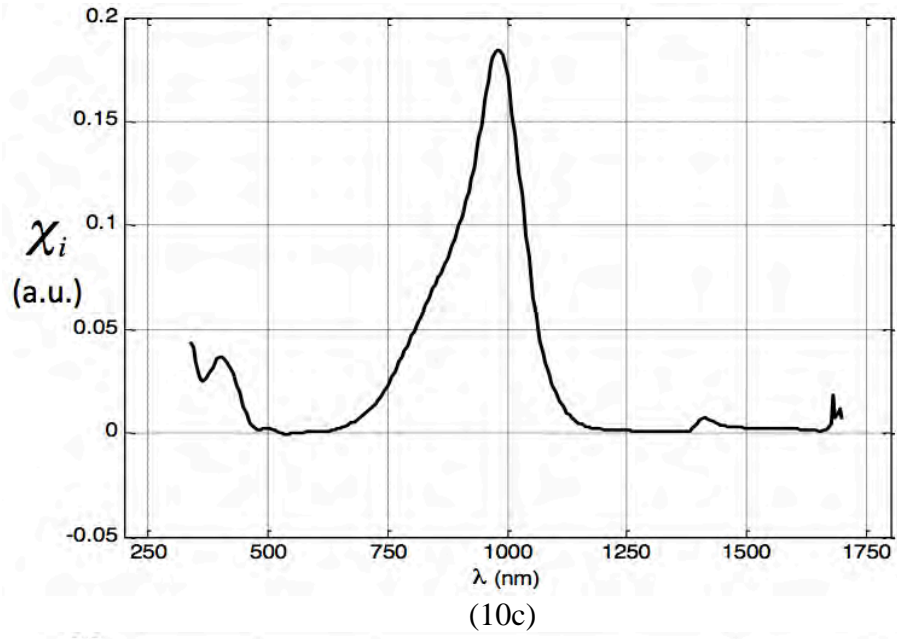
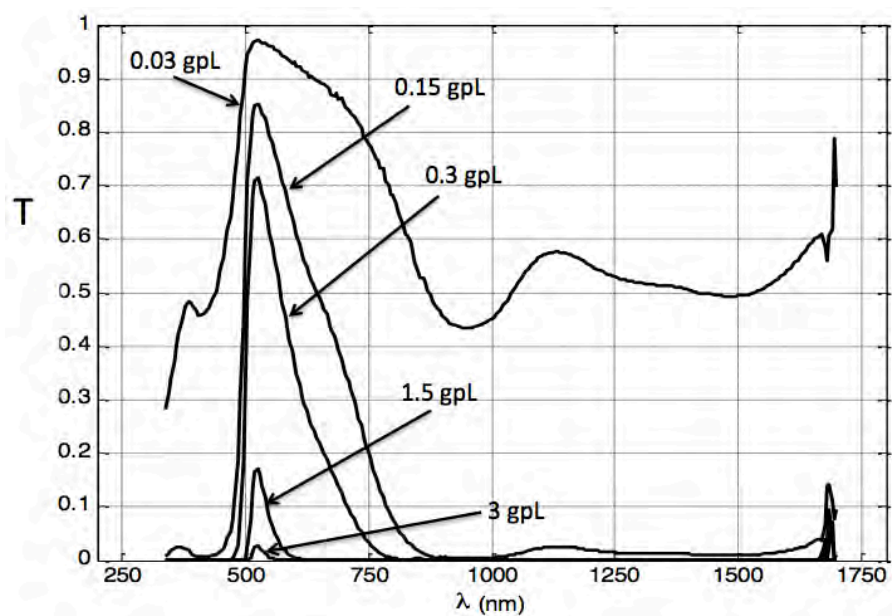
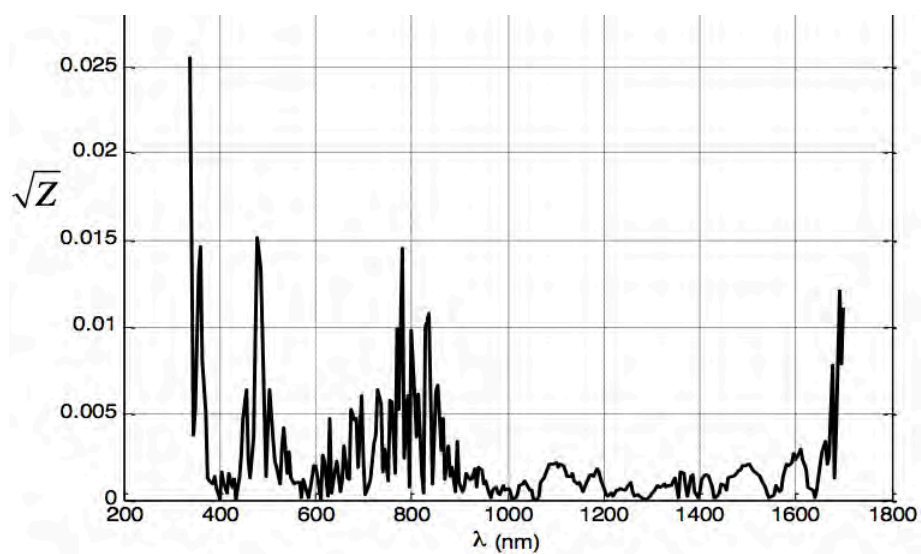


Figure 10. (a) Transmission spectra as a function of triarylamine dye concentration in acetone, where each spectrum was both normalized and background-corrected. (b) Objective function for model parameter optimization with respect to measured transmission spectra for 0.03 gpL triarylamine dye in acetone. (c) Estimated imaginary part of complex susceptibility to within adjustable scale factor for triarylamine dye. (d) Estimated real part of complex susceptibility to within adjustable scale factor for triarylamine dye.



(11a)



(11b)

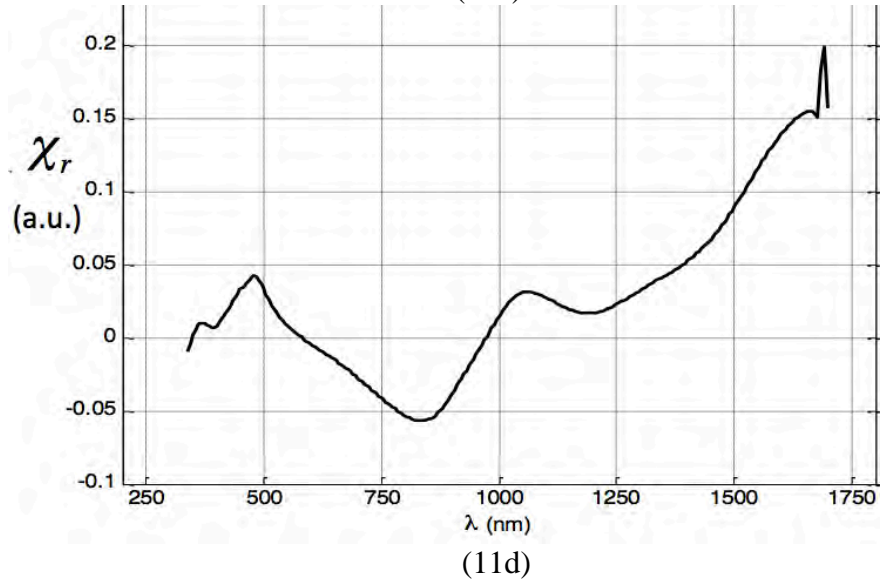
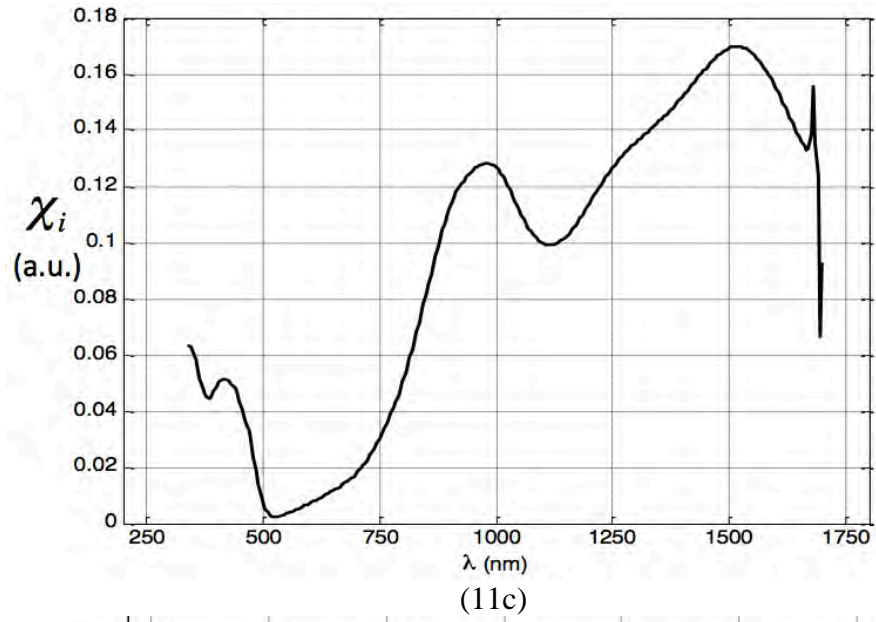
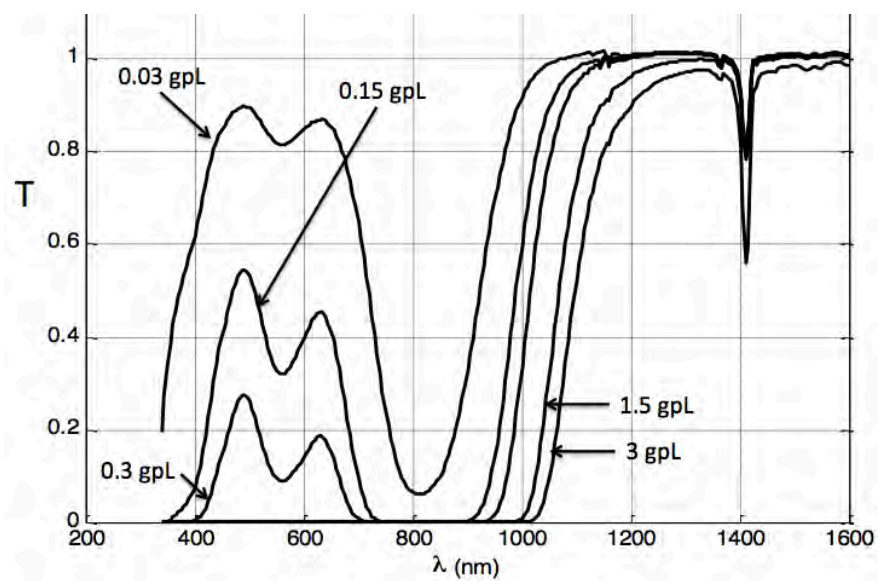
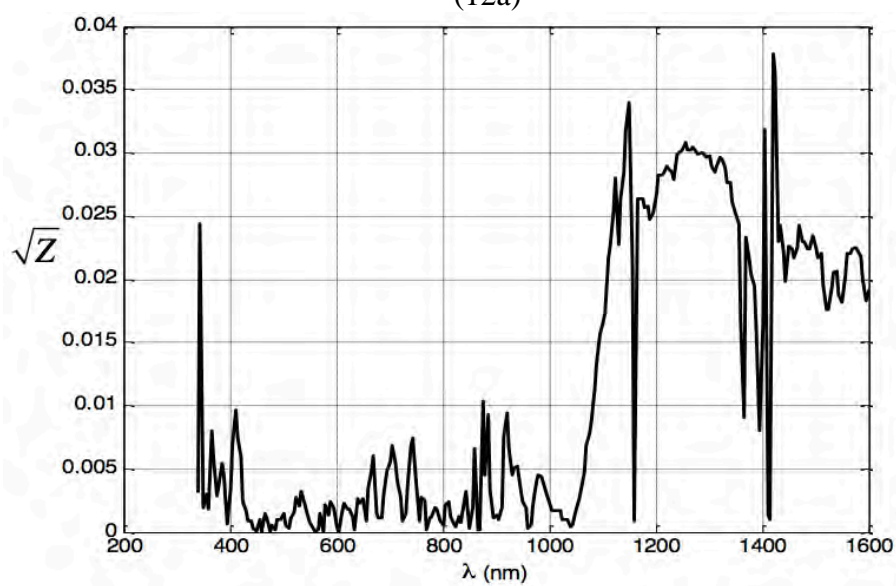


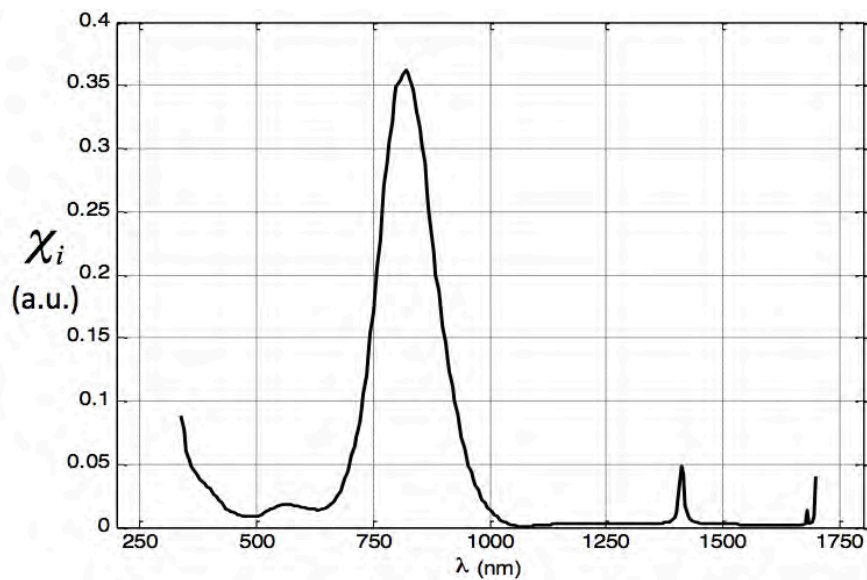
Figure 11. (a) Transmission spectra as a function of tetraaryldiamine dye concentration in acetone, where each spectrum was both normalized and background-corrected. (b) Objective function for model parameter optimization with respect to measured transmission spectra for 0.03 gpL tetraaryldiamine dye in acetone. (c) Estimated imaginary part of complex susceptibility to within adjustable scale factor for tetraaryldiamine dye. (d) Estimated real part of complex susceptibility to within adjustable scale factor for tetraaryldiamine dye.



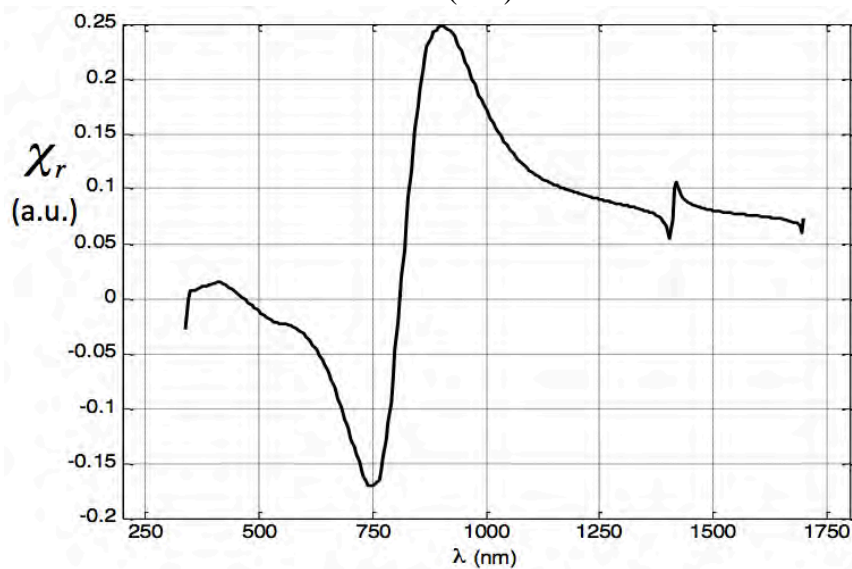
(12a)



(12b)

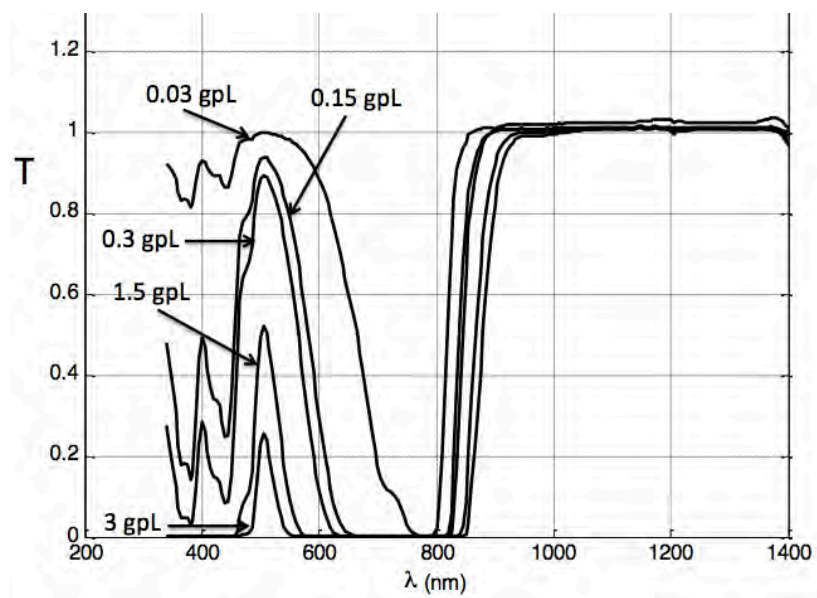


(12c)

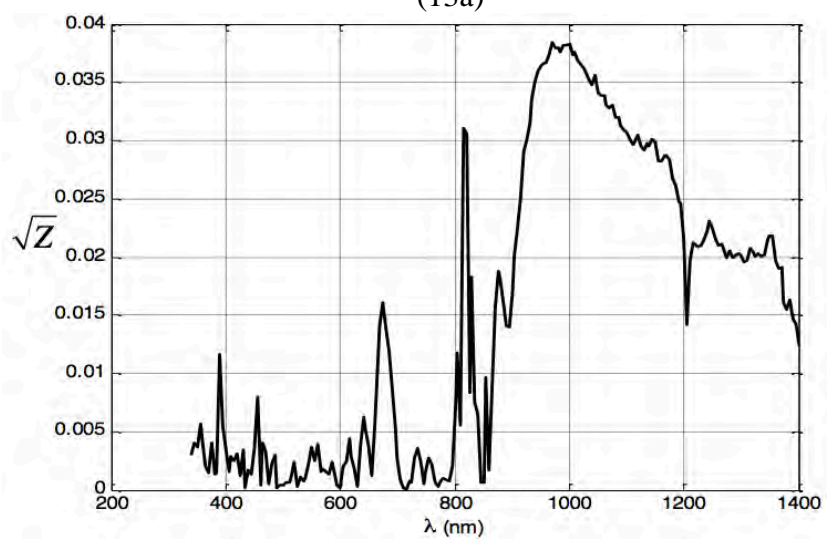


(12d)

Figure 12. (a) Transmission spectra as a function of transition metal dithiolene dye concentration in methylene chloride, where each spectrum was both normalized and background-corrected. (b) Objective function for model parameter optimization with respect to measured transmission spectra for 0.03 gpL transition metal dithiolene dye in methylene chloride. (c) Estimated imaginary part of complex susceptibility to within adjustable scale factor for transition metal dithiolene dye. (d) Estimated real part of complex susceptibility to within adjustable scale factor for transition metal dithiolene dye.



(13a)



(13b)

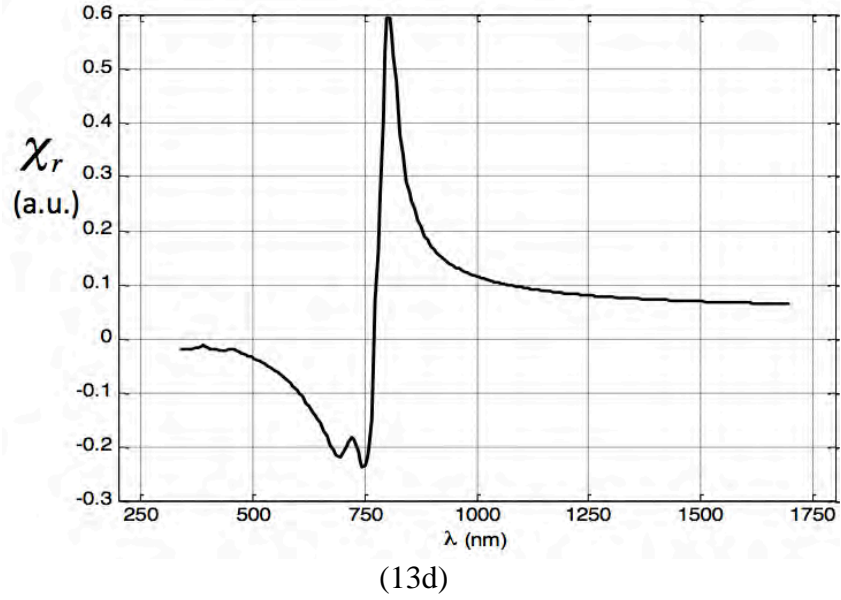
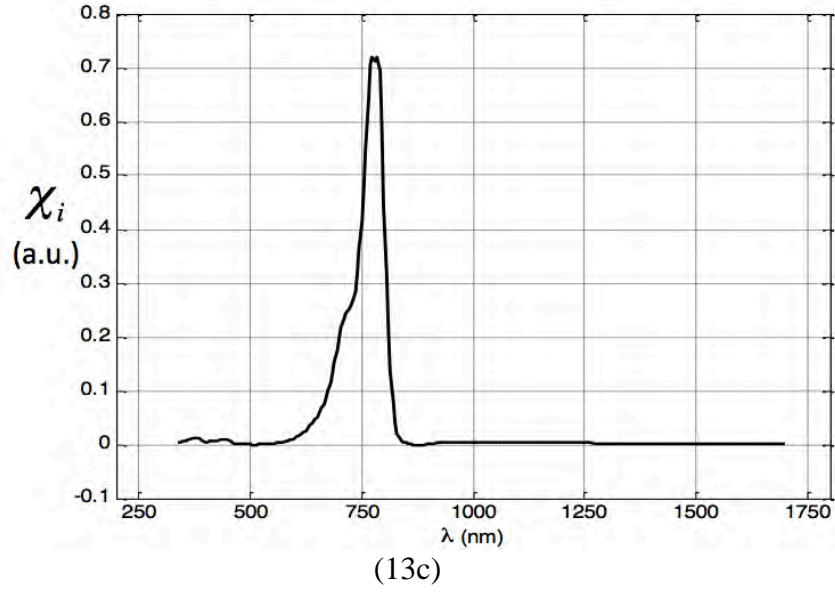


Figure 13. (a) Transmission spectra as a function of indiolium iodide dye concentration in methanol, where each spectrum was both normalized and background-corrected. (b) Objective function for model parameter optimization with respect to measured transmission spectra for 0.03 gpL indiolium iodide dye in methanol. (c) Estimated imaginary part of complex susceptibility to within adjustable scale factor indiolium iodide dye. (d) Estimated real part of complex susceptibility to within adjustable scale factor for indiolium iodide dye.